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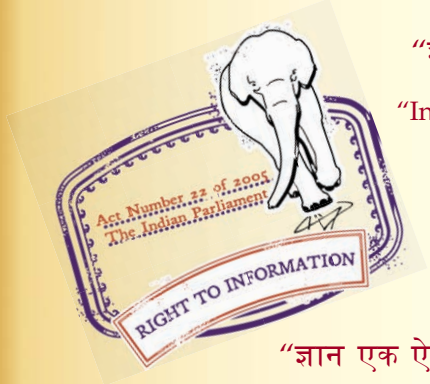

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IS : 3186 - 1965
(Reaffirmed 2006)

Indian Standard
METHODS OF CHEMICAL ANALYSIS OF CADMIUM COPPER

UDC 669·35·73 : 543

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

September 1965

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF CADMIUM COPPER

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Indian Standard
METHODS OF CHEMICAL ANALYSIS OF CADMIUM COPPER

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 1 August 1965, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Cadmium copper is used for making wire for telegraphic and telephone purposes. Suitable methods have been laid down in this standard to determine its chemical composition.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in the country. This has been met by deriving assistance from the 'Book of ASTM Methods of chemical analysis of metals', 1962, published by the American Society for Testing and Materials.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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1. SCOPE

1.1 This standard prescribes methods for determining copper and cadmium in the ranges as specified in IS : 2655-1964†.

2. SAMPLING

2.1 The sample shall be drawn and prepared in accordance with IS : 1817-1961‡.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960§) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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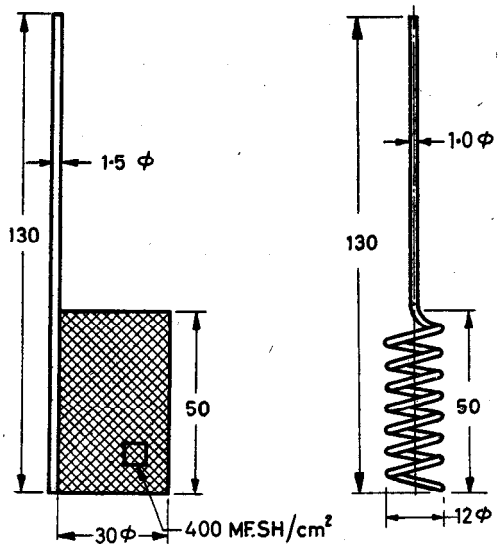
*Rules for rounding off numerical values (*revised*).
†Specification for cadmium copper wire for telegraph and telephone purposes.
‡Methods of sampling non-ferrous metals for chemical analysis.
§Specification for water, distilled quality (*revised*).

4. DETERMINATION OF COPPER BY THE ELECTROLYTIC METHOD

4.1 Outline of the Method — The sample is dissolved in sulphuric acid-nitric acid mixture. Copper is deposited electrolytically from the solution and weighed.

4.2 Apparatus — The following platinum electrodes (*see* Fig. 1) are recommended but strict adherance to the shape and size of the electrodes is not essential. In order to decrease the time of deposition, one of the types of rotating forms of electrodes generally available for agitation of electrolyte may be employed.



1A Cylindrical Platinum Cathode 1B Spiral Platinum Anode

All dimensions in millimetres.

FIG. 1 CYLINDRICAL PLATINUM CATHODE AND SPIRAL PLATINUM ANODE

4.2.1 *Cathode* — It may be formed either from plain or perforated sheet or from wire gauze.

4.2.1.1 Gauze cathodes made preferably from gauze containing 400 mesh/cm² should be used. The wire used for making gauze should be approximately 0·20 mm in diameter. Cathodes should be stiffened by doubling the gauze for about 3 mm on the top and bottom or by reinforcing the gauze at the top and bottom with a platinum ring or band.

4.2.1.2 The diameter and height of the cylinder should be approximately 30 mm and 50 mm respectively. The stem should be made from platinum alloy wire such as platinum-iridium, platinum-rhodium or platinum-ruthenium having diameter of approximately 1·5 mm. It should be flattened and welded to the entire height of the gauze. The overall height of the cathode including the stem should approximately be 130 mm.

4.2.2 *Anode* — Either a spiral or a gauze anode should be used. The spiral anode should be made from 1·0 mm or larger platinum wire formed into a spiral of seven coils with a height of approximately 50 mm and diameter of 12 mm, the overall height including the stem being 130 mm. The gauze anode should be made of the same material and of the same general design as platinum gauze cathode specified under **4.2.1**.

4.3 Reagent

4.3.1 *Sulphuric Acid-Nitric Acid Mixture* — Add slowly, with constant stirring, 300 ml of concentrated sulphuric acid (sp gr 1·84; conforming to IS : 266-1961*) to 750 ml of water, cool, add 210 ml of concentrated nitric acid (sp gr 1·42; conforming to IS : 264-1950†).

4.3.2 *Ethanol or Methanol* — 95 percent (*v/v*).

4.4 Procedure

4.4.1 Transfer 2 g of an accurately weighed sample into a 250-ml beaker provided with a cover glass and add 20 ml of sulphuric acid-nitric acid mixture. After the initial reaction is over, heat to 80° to 90°C until completely dissolved and brown fumes and expelled.

4.4.2 Wash down the cover and sides of the beaker and dilute the solution to 200 ml. Add 2 g of urea, insert the electrodes, the cathode having been accurately weighed; cover with a pair of split cover glass and electrolyze for 16 hours at a current density of 0·6 A/dm^2 (at this current density, the electrolysis is conveniently carried on overnight), or at a current density of 4 A/dm^2 for a short period (about 2·5 hours). In the latter case, one of the types of rotating forms of electrodes generally available may be used. When the solution becomes colourless reduce the current density to 0·3 A/dm^2 and continue electrolysis until the deposition of copper is complete as indicated by absence of plating on the new surface of the electrode obtained by raising the level of the solution.

4.4.3 Without interrupting the current, raise the electrode assembly. While raising, rinse thoroughly with water and collect the washings into the electrolyte. Remove the cathode quickly while washing further with

*Specification for sulphuric acid (*revised*).
†Specification for nitric acid.

water; rinse it with water in a beaker and then dip it in two successive baths of ethanol or methanol. Dry the cathode in an air oven at 110°C for three to five minutes, cool and weigh for copper. Preserve the electrolyte for the determination of cadmium.

4.5 Calculation

$$\text{Copper, percent} = \frac{A}{B} \times 100$$

where

A = weight in g of copper, and

B = weight in g of the sample taken.

5. DETERMINATION OF CADMIUM BY EDTA (VOLUMETRIC) METHOD

5.1 Outline of the Method — Cadmium is precipitated from the electrolyte preserved under **4.4.3** by diethyldithiocarbamate; dissolved in hydrochloric acid and determined volumetrically by titration with EDTA.

5.2 Reagents

5.2.1 *Tartaric Acid Solution* (300 *g/l*) — Dissolve 300 g of tartaric acid in 700 ml of water with heat, cool to room temperature, and dilute to one **litre.**

5.2.2 *Methyl Red Indicator Solution* (0·4 *g/l*) — Dissolve 0·1 g of methyl red in 3·72 ml of 0·100 0 N Sodium hydroxide solution and dilute to 250 ml with water. Filter if necessary.

5.2.3 *Sodium Hydroxide Solution* (200 *g/l*) — Dissolve 200 g of sodium hydroxide in 800 ml of water, cool to room temperature, and dilute to one litre.

5.2.4 *Potassium Cyanide* (100 *g/l*) — Dissolve 100 g of potassium cyanide in 500 ml of water and dilute to one litre.

CAUTION — The preparation, storage, and use of potassium cyanide solutions requires care and attention. Avoid inhalation of fumes and exposure of skin to the chemical or its solutions. Work in a well-ventilated room.

5.2.5 *Sodium Diethyldithiocarbamate Solution* (20 *g/l*) — Dissolve 20 g of the salt in 800 ml of water with heat, cool to room temperature, and dilute to one litre.

5.2.6 *Wash Solution* — To 20 ml of sodium hydroxide solution add 10 ml of potassium cyanide solution and 10 ml of sodium diethyldithiocarbamate solution and dilute to one litre.

5.2.7 *Concentrated Hydrochloric Acid* — sp gr 1·16 (conforming to IS : 265-1962*).

5.2.8 *Buffer Solution* — Dissolve 54 g of ammonium chloride in 300 ml of water, add 350 ml of ammonium hydroxide and dilute to one litre. The pH value of this solution is 10.

5.2.9 *Eriochrome Black-T Indicator Solution* — Dissolve 0·4 g of Eriochrome black-T1- (1-hydroxy-2-naphtholazo) -5-nitro-2-naphthol-4-sulphonic acid, sodium salt in 20 ml of ethanol. Add 30 ml of triethanolamine and store in a polythene dropping bottle.

5.2.10 *Formaldehyde* (1 : 9) — Dilute 100 ml of formaldehyde (37 percent) with 900 ml of water.

5.2.11 *Standard Disodium Ethylenediamine Tetra-acetate (EDTA) Solution* (0·025 *M*) — Dissolve 9·6 g of the salt in 600 ml of water while heating. Cool to room temperature, add 0·1 g of magnesium chloride ($MgCl_2.6H_2O$), and dilute to one litre with water. Standardize the solution as follows.

5.2.11.1 Transfer, to an 800-ml beaker, an aliquot of the standard zinc solution. Continue as directed in **5.3.5**. Calculate the equivalent of the EDTA solution in terms of grams of zinc per millilitre of solution.

5.2.12 *Standard Zinc Solution* (1 *ml* = 0·001 0 *g of Zn*) — Dissolve 1·000 g of pure zinc in 50 ml of sulphuric acid (1 : 4), cool to room temperature, and dilute to one litre.

5.3 Procedure

5.3.1 Evaporate the electrolyte reserved under **4.4.3**. Cool to room temperature and dilute to 250 ml with water.

5.3.2 Add 15 ml of tartaric acid solution and 8 drops of methyl red indicator. Carefully neutralize the solution with sodium hydroxide solution avoiding excess. Add 10 ml of potassium cyanide solution and 75 ml of sodium diethyldithiocarbamate solution. Stir thoroughly and allow to stand for about 30 minutes.

5.3.3 Filter the solution with suction through the fritted glass crucible. Wash the precipitate twice by decantation with the wash solution (*see* **5.2.6**). Transfer the crucible and the precipitate to a 250-ml beaker and add 25 ml of concentrated hydrochloric acid and 30 ml of water to completely cover the precipitate and the crucible. Heat until the precipitate is completely dissolved. Cool to room temperature and dilute to 200 ml.

5.3.4 Neutralize the solution with sodium hydroxide solution using methyl red indicator. Add 30 ml of buffer solution, 10 ml of potassium

*Specification for hydrochloric acid (*revised*).

cyanide solution, 5 drops of eriochrome black-T indicator and sufficient formaldehyde to just give a red colour.

5.3.5 Titrate slowly with standard EDTA solution to a bluish-green end point. Add 5 ml of formaldehyde solution and if the colour changes to red, titrate again with EDTA solution to a bluish-green end point. Continue the formaldehyde addition, and if necessary, the EDTA titrations, until the bluish-green end point is stable for at least two minutes for the last formaldehyde addition.

5.4 Calculation

$$\text{Cadmium, percent} = \frac{A \times B}{C} \times 100$$

where

A = volume in ml of EDTA solution required for titration of the solution,

B = cadmium equivalent in g/ml of EDTA solution, and

C = weight in g of the sample taken.

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